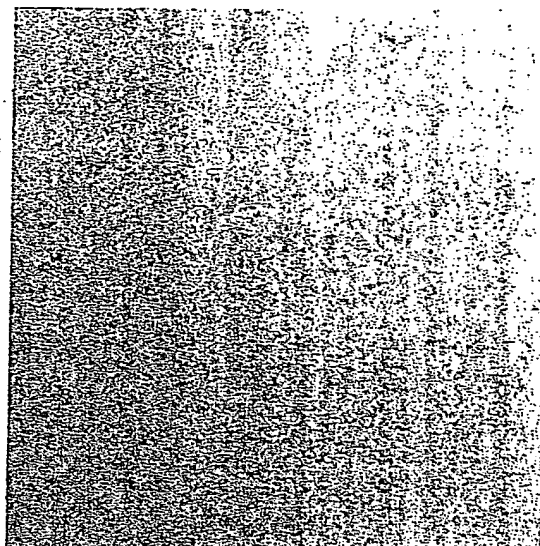


May. 28. 2004 3:10PM

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No. 0970 P. 2

Japanese Kokai Patent Application No. Sho 56[1981]-53602



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Job No.: 391-87568

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JAPANESE PATENT OFFICE  
PATENT JOURNAL (A)  
KOKAI PATENT APPLICATION NO. SHO 56[1981]-53602

Int. Cl.<sup>3</sup>: A 01 N 25/08  
Sequence No. for Office Use: 6742-4H  
Filing No.: Sho 54[1979]-129337  
Filing Date: October 7, 1979  
Publication Date: May 13, 1981  
No. of Inventions: 1 (Total of 3 pages)  
Examination Request: Not filed

SLIME REMOVAL AGENT

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[There are no amendments to this patent.]

Claims

1. A type of slime removal agent characterized by the fact that it is prepared by adsorbing one or several compounds having physiological activity for slime, such as organic halogen compounds, organic quaternary ammonium salts, organic heterocyclic compounds, organic nitrogen compounds, organic sulfur compounds, salts of nitrogen-containing acids, etc., on a porous inorganic carrier.

2. The slime removal agent described in Claim 1, characterized by the fact that the porous inorganic carrier is petaline [sic] calcium silicate and/or petaline silica made of flake-shaped aggregate.

Detailed explanation of the invention

This invention pertains to a powdery type of slime removal agent that can be easily dispersed in water. More specifically, this invention pertains to a powdery type of slime removal agent that can be easily dispersed in water and is prepared by adsorbing one or several compounds with physiological activity for slime on a porous inorganic carrier, in particular, on petaline calcium silicate and/or petaline silica.

In the slime removal agent of this invention, compounds with physiological activity for slime are adsorbed on petaline calcium silicate and/or petaline silica, and it is characterized by the fact that no surfactant or other dispersion aid is used.

In the prior art, as far as the form of the formulation of slime removal agent is concerned, it is usually in liquid form. For preparing the liquid formulation, solvents, such as dimethyl formamide, glycol-based compounds, polyalkylene glycol-based compounds, etc., are used. Usually, the concentration of chemicals in the liquid formulation is about 10 wt%. When the liquid formulation is diluted with water, it is dissolved to form a nearly transparent solution. Consequently, after use, almost all of the organic material contained in the liquid formulation is contained in the discharge water. As a result, COD is increased, and it becomes expensive to treat the wastewater.

In order to solve the aforementioned problems, the present inventors have performed extensive research. As a result of this research, it was found that for the powder prepared by adsorbing compounds having physiological activity for slime on petaline calcium silicate and/or petaline silica, even when no surfactant is used, it still can be uniformly dispersed in water, and there is no increase in the physiological activity for slime. In addition, by filtering the discharge water after use, it is possible to remove almost all of the organic material. In this way, this invention was reached.

According to this invention, as far as petaline calcium silicate and/or petaline silica used as porous inorganic carrier are concerned, the former has a gyrolite-type cylindrical structure, and the latter is amorphous. Both are flake-shaped aggregates in appearance, with an apparent specific volume as large as 5-25 cc/g and with a large pore volume. They are both manufactured by Tokuyama Soda Co., Ltd. The former has a commercial name of Flolite [transliteration] R, and the latter has a commercial name of Flolite S-700. Also, because this adsorbent has a larger pore size than that of other organic powders, it is the best material for use as carrier in adsorbing compounds with relatively high molecular weight at a high concentration. Because the maximum adsorptivity is 800 cc/100 g, when compounds with physiological activity for slime are adsorbed, it is preferred that the adsorbed amount be lower than said maximum adsorptivity. When the active chemical is in liquid form, it can be adsorbed as it is. However, when the active chemical is in solid form, it is dissolved at the maximum concentration in a solvent that can dissolve it

well, followed by adsorption. In order to prevent an increase of COD in discharge water, it is preferred that a solvent that is poorly soluble in water be used.

For the slime removal agent prepared above, when it is simply added to water and agitated, a uniform dispersion can be formed, and the disperse state can be maintained for a long time.

The slime removal agent of this invention can be used at any site where slime may occur during the papermaking operation. Because petaline calcium silicate and/or petaline silica have excellent freeness, when it is contained in paper in the papermaking process, it has no adverse influence on the substance of the paper [sic; on the properties of the paper]. On the contrary, it may be used as a material that can be added actively as a paper modifier.

According to this invention, any of the compounds with physiological activity for slime may be used. The following is a list of some typical examples of the compounds:

2-bromo-2-nitroethanol, 2-bromo-2-nitrobutanol, 1-(p-nitrophenyl)-2-bromo-2-nitrobutanol, bis(bromoacetoxy)butene, bis(bromoacetoxy)butane, n-octyl ester of bromoacetic acid, n-lauryl ester of bromoacetic acid, dibromodimethyl hydantoin, amine salt of bromoacetic acid, chlorinated isocyanuric acid, lauryl benzylammonium chloride, oleyl benzylammonium chloride, methyl isothiazole, chloromethyl isothiazole, benzisothiazolinone, amine salt of ethylene-bis(dithiocarbamate), methylene-bis(thiocyanate), 2-chloro-4,6-bis(ethylamino-s-triazine), 2-methylthio-4,6-bis(ethylamino-s-triazine), etc.

These compounds may be used either alone or as a mixture of several types to ensure good effect without failure in any case.

In the following, this invention will be explained in detail with reference to application examples. However, this invention is not limited to these application examples.

#### Application Example 1

200 parts by weight of 2-bromo-2-nitrobutanol were uniformly adsorbed on 100 parts by weight of Flolite R100 manufactured by Tokuyama Soda Co., Ltd. to form a powder of slime removal agent that can be easily dispersed in water. The powder has a nearly white color.

The slime removal agent was tested using the following method.

Test method: Using the agar dilution method, culturing was performed at 30°C for 48 h, and the minimum inhibitory concentration of the slime removal agent was measured.

Results of the test indicated that for *Aspergillus niger*, growth was inhibited at 4 ppm. For *Escherichia coli*, growth was inhibited at 21 ppm.

Test of influence on paper:

The pulp concentration was adjusted to 3%. 0.5% (solids) of a sizing agent (commercial name: Sizebire E [transliteration]) was added, and the pH was adjusted to 4.0 with aluminum

sulfate. After agitation for 10 min, it was diluted 10X with water. Then, the slime removal agent was added at concentrations listed in Table 1. After agitation for 10 min, papermaking was performed on a sheet machine. It was found that the freeness was good in the all of the tests. Test of paper quality was performed using the conventional method, with the following results obtained.

Table 1

Concentration	0	30	50	100	150
Test item					
Whiteness (P-8113)	70.4	70.3	70.2	70.3	70.5
Tensile strength (P-8112)	3.91	3.91	3.90	3.90	3.91
Rupture strength (P-8116)	14	15	15	14	14
Size degree sec (P-8122)	12.1	12.2	12.6	12.1	12.3

As listed above, the slime removal agent of this invention displays an inhibiting effect on slime. Also, it has no adverse influence on the paper quality.

#### Application Example 2

The chemicals listed in Table 2 were adsorbed at a ratio by weight of 60% with respect to Flolite R to form a powder of slime removal agent. For each sample, the minimum inhibitory concentration was tested using the agar diluting method in Application Example 1. As can be seen from Table 2, the slime removal agent of this invention has an excellent effect of inhibiting slime.

Table 2

Name of chemical	Name of bacteria	
	<i>Aspergillus niger</i>	<i>Escherichia coli</i>
bis(bromoacetoxy)butene	2 ppm	5 ppm
1,3-dibromo-5,5-dimethyl hydantoin	1 ppm	1.5 ppm
n-lauryl ester of bromoacetic acid	3 ppm	8 ppm
5-chloro-2-methyl-4-isothiazolone-3-one [sic; ...-isothiazol-3-one]	2 ppm	7 ppm
2-methyl-4-isothiazolone-3-one	15 ppm	19 ppm
p-lauryl benzylammonium chloride	10 ppm	18 ppm
Methylene-bis(thiocyanate)	0.5 ppm	1 ppm
2-chloro-4,6-bis(ethylamino-s-triazine)	21 ppm	27 ppm
Benzisothiazolone-2-one	3 ppm	8 ppm

Application Example 3

30 parts by weight of bis(bromoacetoxy)butene were uniformly adsorbed on 10 parts by weight of Flolite S-700 manufactured by Tokuyama Soda Co., Ltd. to form a slime removal agent.

During a papermaking operation, the slime removal agent of this invention was added to the rifler at 200 ppm once every 6 h, and the state of generation of slime was observed during a period of 30 days. Little slime was observed during this period. The result was good.

(1a)

⑬ 日本国特許庁 (JP)

⑪ 特許出願公開

⑫ 公開特許公報 (A)

昭56—53602

⑤ Int. Cl.<sup>3</sup>  
A 01 N 25/08

識別記号

庁内整理番号  
6742—4H

④ 公開 昭和56年(1981)5月13日

発明の数 1  
審査請求 未請求

(全 3 頁)

④ スライム除去剤

光市新幸町56番地

① 特 願 昭54—129337

⑦ 出 願 人 株式会社千代田化学研究所  
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番3号

② 出 願 昭54(1979)10月7日

③ 発 明 者 前田昭朗

明 細 書

1 発明の名称 スライム除去剤

2 特許請求の範囲

① 有機ハロゲン化合物、有機第4級アンモニウム塩、有機ヘテロ環化合物、有機窒素化合物、有機硫黄化合物、窒化酸塩など、スライムに対し生理活性を有する化合物の1種ないし2種以上を多孔性無機担体に吸着させてなるスライム除去剤。

② 多孔性無機担体が薄片状の集合体からなる花卉状ケイ酸カルシウムおよび/または花卉状シリカである特許請求の範囲①記載のスライム除去剤。

3 発明の詳細な説明

本発明は、粉末状で水易分散性のスライム除去剤に関する。更に詳しくいえば、スライムに対し生理活性を有する化合物の1種ないし2種以上を多孔性無機担体、特に花卉状ケイ酸カルシウムおよび/または花卉状シリカに吸着させてなる粉末状で水易分散性のスライム除去剤に関するものである。

(1)

本発明のスライム除去剤は、花卉状ケイ酸カルシウムおよび/または花卉状シリカに、スライムに対し生理活性を有する化合物を高濃度に吸着させてなるものであり、界面活性剤などの分散助剤を使用しないところに特徴がある。

従来、スライム除去剤の製剤の形態としては、液剤が最も多く、これら液剤はジメチルホルムアミド、グリコール系化合物、ポリアルキレングリコール系化合物などの溶剤を使用している。液剤中の薬剤は、10重量%前後の濃度としているものが多い。液剤を水で希釈すると、ほぼ透明に溶解し、従って使用後、液剤に含まれる有機物のほとんどすべてのものは、排水中に含まれてくるため、CODを増加させる原因となり、废水处理に多大の経費がかかるなどの欠点があった。

本発明は、これらの欠点を改良すべく研究を重ねた結果、スライムに対し生理活性を有する化合物を花卉状ケイ酸カルシウムおよび/または花卉状シリカに吸着させた粉体が、界面活性剤を使用しないにも係らず、水に均一分散し、スライム

(2)

表 1

試験項目 (JIS)	添加濃度 (ppm)	0	30	50	100	150
白色度 % (P-8113)		70.4	70.3	70.2	70.3	70.5
引張強度 $K_p$ (P-8112)		3.91	3.91	3.90	3.90	3.91
引張強度 $K_p$ (P-8116)		14	15	15	14	14
サイズ度 秒 (P-8122)		12.1	12.2	12.6	12.1	12.3

以上の如く、本発明のスライム除去剤は、スライムに対して阻止効果があり、しかも紙質に対しては何ら悪影響を及ぼさなかった。

## 実施例 2

表 2 に示した薬剤をフローライト B に対し、重量比で 60 % になるように吸着させて、スライム除去剤粉末を得た。各々につき実施例 10 の凍天希釈法によって最低発育阻止濃度を試験した。表 2 で明らかなように、本発明の除去剤は優れた阻止効果を示した。

(7)

除去剤を 200 ppm あて 6 時間おきに投入し、30 日間にわたってスライムの発生状況を観察した。この間、スライムの発生はほとんど見られず良好な結果を得た。

特許出願人

株式会社 千代田化学研究所

表 2

薬剤名	商 名	アスメルゼル ス・ニブル	エシロシレ・コラ
ビス(プロモアセチル)ブテン		2 ppm	5 ppm
1,3-ジプロモ-5,5-ジフルオロシクロペンタン		1 ppm	1.5 ppm
プロモ酢酸-ローラクリルエステル		3 ppm	8 ppm
5-ブromo-3-フルオロ-4-イソシアロノ-3-オン		2 ppm	7 ppm
2-メチル-4-イソシアロノ-3-オン		15 ppm	19 ppm
ト-ブアル-10-フル-2-メチル-ブレン		10 ppm	18 ppm
メチレンビス(チオレブネート)		0.5 ppm	1 ppm
2-ブromo-4,6-ビス(チオレブネート)ブレン		21 ppm	27 ppm
メンスイソシアロノ-3-オン		3 ppm	8 ppm

## 実施例 3

徳山曹達(株)製フローライト B-700。

10 重量部にビス(プロモアセチル)ブテン 30 重量部を均一に吸着させて、スライム除去剤を得た。

製紙工程におけるリフラーに本発明のスライム

(8)

(9)

# EUROPEAN PATENT OFFICE

## Patent Abstracts of Japan

PUBLICATION NUMBER : 56053602  
PUBLICATION DATE : 13-05-81

APPLICATION DATE : 07-10-79  
APPLICATION NUMBER : 54129337

APPLICANT : CHIYODA KAGAKU KENKYUSHO:KK;

INVENTOR : MAEDA AKIO;

INT.CL : A01N 25/08

TITLE : REMOVING AGENT FOR SLIME

ABSTRACT : PURPOSE: The titled powdery removing agent dispersing in water readily without using a dispersing agent, e.g., surface active agent, etc., obtained by adsorbing a compound, e.g., organic halogen compound, organic quaternary ammonium salt, etc. having physiological activity to slime on a porous inorganic carrier.

CONSTITUTION: One or two or more of compounds selected from the group consisting of an organic halogen compound, organic quaternary ammonium salt, organic heterocyclic compound, organic nitrogen compound, organic sulfur compound, salt of nitrogenized acid, etc. having physiological activity to slime are adsorbed on a porous inorganic carrier, e.g., petaline calcium silicate and/or petaline silica in the form of laminar aggregate to give a removing agent for slime. The removing agent is only added to water, and it can be dispersed in water uniformly and keep dispersing state for a long time. It can be applied at any place where slime occurs especially in paper producing process. Even if it is included in paper directly, it has no bad influence on the quality of paper at all and can be used as a paper modifier.

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